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DATA EVALUATION RECORD

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Merricks, D.L. 1989j. Metolachlor field dissipation study in Iowa-Site B. Agrisearch Project No. 1265-B. Unpublished study performed by Agrisearch Incorporated, Frederick, MD, and submitted by Ciba-Geigy Corporation, Greensboro, NC.

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CONCLUSIONS:

Field Dissipation - Terrestrial

1. This study is considered supplemental at present.
2. This study will be reevaluated upon receipt of an explanation of the following:

The results from the laboratory fortified control samples, fortified at 0.05 to 5.0 ppm (all degradates) or to 10.0 ppm (metolachlor), carried through the procedure with the test samples indicate a wide variation in the recovery efficiencies. These recovery efficiencies ranged from 55 to 175% of the applied for metolachlor, 53 to 132% for CGA-51202, 42 to 150% for CGA-40172, 52 to 223% for CGA-40919, and 42 to 199% for CGA-50720 (Table 7).

The degradates CGA-40172 and CGA-40919 were included in this field dissipation study. In the aerobic and anaerobic soil metabolism study (MRID # 41309801-B) under aerobic conditions these two degradates were only detected but not quantifiable. However, other degradates (CGA-41638 was 2.06% of applied at 90 days, CGA-37735 was 1.27% at 30 days, and CGA-13656 was 1.02% immediately posttreatment) were not also included or used in place of degradates that occurred at such minimal levels that they could only be detected but not quantified (See Comment # 3).

3. Metolachlor dissipated with a registrant calculated half-life of 117 days from the upper 6 inches of a bareground silty clay loam soil field plot treated with metolachlor (28% G) at 6 lb ai/A. In an adjacent plot that was treated with metolachlor at 4.0 lb ai/A and then immediately planted to beans, metolachlor dissipated with a registrant-calculated half-life of 144 days from the 0-to 6-inch soil layer.

METHODOLOGY:

Metolachlor (Dual 25G, 28% G, Ciba-Geigy) was surface-applied by hand, using a broadcast spreader, at 4 and 6 lb ai/A to two field plots (50 x 50 feet) of silty clay loam soil (0- to 6-inch layer; 16% sand, 54% silt, 30% clay, 3.3% organic matter, pH 6.8, CEC 18.3 meq/100 g) located in Des Moines, Iowa, on June 4, 1987. One week prior to application, the field had been disked to a depth of 12 inches. The day before treatment, the plots were disked twice to a 6-inch depth; immediately following treatment, the test substance was incorporated by disking. The plot treated at 4 lb ai/A was planted to beans immediately posttreatment; the plot treated at 6 lb ai/A was left bare. Untreated bareground and bean plots (sizes unspecified) located 150 feet upslope were maintained as controls (Figure 3). Twenty soil cores (5-foot length, 1-inch diameter, stainless steel probe with an acetate liner) were collected prior to treatment; four cores per subplot from three randomly selected subplots were taken at each sampling interval between 0 and 546 days posttreatment. Cores were stored frozen for up to 698 days prior to extraction (Appendix D).

Frozen soil cores were divided into 6-inch increments, and the increments from the four cores per subplot were composited and homogenized. A portion of each composited sample (50 g) was refluxed with methanol: water (1:1) for 1 hour. An aliquot (60 mL) of the extract was mixed with a water:saturated sodium chloride solution, the pH was adjusted to 1-1.5 with 1 N sulfuric acid, and the solution was then partitioned three times with hexane:ethyl acetate (1:1, v:v). The organic phases were combined and dried with anhydrous sodium sulfate; the solution was then methylated with diazomethane. After 30 minutes, the solution was evaporated to dryness, redissolved in hexane, and analyzed for metolachlor and its degradates CGA-51202, CGA-40172, CGA-40919, and CGA-50720 by GC using an OV-17 capillary column and a flame-thermionic detector. The detection limits were 0.05 ppm for metolachlor, CGA-51202, and CGA-40172; 0.06 ppm for CGA-40919; and 0.07 ppm for CGA-50720. Recovery efficiencies from soil samples fortified at 0.05 to 5.0 ppm (all degradates) or to 10.0 ppm (metolachlor) ranged from 55 to 175% of the applied for metolachlor, 53 to 132% for CGA-51202, 42 to 150% for CGA-40172, 52 to 223% for CGA-40919, and 42 to 199% for CGA-50720 (Table 7). The concentrations of metolachlor and its degradates detected in the field soil samples were corrected for recoveries that were <100%.

Selected soil samples were also analyzed for metolachlor and its degradates by GC/MS using single ion monitoring; the detection limit was 0.1 ng/uL.

DATA SUMMARY:

Metolachlor dissipated with a registrant-calculated half-life of 117 days ($R^2 = 0.752$) from the upper 6 inches of a bareground field plot (50 x 50 feet) of silty clay loam soil in Des Moines, Iowa, that was treated with metolachlor (25% G) at 6 lb ai/A on June 4, 1987. In the 0- to 6-inch soil depth, average metolachlor concentrations ranged from 0.55 to 1.96 ppm between 0 and 15 days posttreatment (maximum 14.5 ppm at 1 day), then decreased to <0.05 ppm by 421 days (final sampling interval at 534 days, Table 2).

In an adjacent plot that was treated with metolachlor at 4.0 lb ai/A and then immediately planted to beans, metolachlor dissipated with a registrant-calculated half-life of 144 days ($R^2 = 0.773$) from the 0- to 6-inch soil layer. In the 0- to 6-inch soil depth, metolachlor was detected at an average of 1.53 ppm immediately posttreatment (maximum 4.18 ppm at 7 days), 0.43 ppm at 3 days, 1.22 ppm at 7 days, 0.49 ppm at 28 days, and <0.05 ppm at 534 days (Table 1).

The concentrations of metolachlor degradates detected in the soil were similar for the bareground and beans plots. In the 0- to 6-inch soil depth, the degradate

CGA-51202

was a maximum of 0.49 ppm at 61 days posttreatment;

CGA-40172

was a maximum of 0.30 ppm at 1 day; and

CGA-40919

was a maximum of 1.12 ppm at 61 days (Tables 11 and 12).

Downward movement of metolachlor resulted in maximum concentrations of 0.28 ppm in the 6- to 12-inch depth, <0.05 ppm in the 12- to 18-inch depth, 0.22 ppm in the 18- to 24-inch depth, and ≤ 0.09 ppm below 24 inches (up to 48 inches). In the 6- to 12-inch soil layer, CGA-51202 was detected at a maximum of 0.21 ppm, CGA-40172 at 0.14 ppm, and CGA-40919 at 0.34 ppm. From 12-36 inches (6-inch increments), the three degradates were detected at ≤ 0.1 ppm, and none was detected (<0.06 ppm) in the 36- to 48-inch soil layer. The degradate CGA-50720 was not detected (<0.07 ppm) in any soil sample at any interval.

During the study, rainfall totaled 49.5 inches, air temperatures ranged from -19 to 102° F, and soil temperatures (0- to 8-inch depth) ranged from 3 to 114° F.

COMMENTS:

1. It appears that the analytical method was unreliable, especially when approaching the limit of detection; recovery from fortified samples was unusually low and variable. Recovery efficiencies from spiked samples for metolachlor at 0.05 ppm ranged from 76 to 166% of the applied, and at 0.1-10.0 ppm ranged from 55 to 175%; for CGA-51202 at

0.05 ppm, recovery efficiencies ranged from 64 to 132%, and at 0.1-5.0 ppm ranged from 53 to 130%; for CGA-40172 at 0.05 ppm, recovery efficiencies ranged from 53 to 143%, and at 0.1-5.0 ppm ranged from 42 to 150%; for CGA-40919 at 0.05 ppm, recovery efficiencies ranged from 52 to 137%, and at 0.1-5.0 ppm ranged from 54 to 223%; for CGA-50720 at 0.05 ppm, recovery efficiencies ranged from 50 to 199%, and at 0.1-5.0 ppm ranged from 42 to 144% (Table 7). The concentrations of metolachlor and its degradates detected in the field soil samples were corrected for recoveries that were <100%.

2. The data were variable such that it is difficult to accurately assess the dissipation of metolachlor and its degradates in the soil. In addition to the variability of average concentrations of metolachlor in the soil between sampling intervals, metolachlor residues were also highly variable from sample to sample at the same interval. For example, in samples from the 0- to 6-inch soil layer taken at day 0, metolachlor ranged from <0.05 to 3.04 ppm in the crop plot (Table 11) and from <0.05 to 7.46 ppm in the bareground plot (Table 12). The variability of the field data may have been due to the inability of the method to accurately determine metolachlor residues (see Comment 1).
3. In the aerobic and anaerobic metabolism study (MRID No. 41309801-B) the major degradates of metolachlor under aerobic conditions were: CGA-51202, reaching a maximum of 28.09% of the applied at 90 days posttreatment; CGA-50720, at a maximum of 14.85% of applied at 272 days; CGA-41638, at a maximum of 2.06% at 90 days; CGA-37735, at a maximum of 1.27% at 30 days; CGA-13656, at a maximum of 1.02% immediately posttreatment. Other degradates that were detected but not quantifiable were CGA-40172, CGA-41507, CGA-40919, and CGA-37913.

In the anaerobic metabolism portion of the same study the major degradate in the soil and flood water was CGA-51202 at a maximum of 23.33% of the applied at 29 days after anaerobic conditions were established. Other degradates isolated from the soil and water were: CGA-41638, at a maximum of 8.30% of the applied at 60 days; CGA-50720, at a maximum of 7.34% at 60 days; CGA-13656, at a maximum of 1.46% at 29 days; and CGA-37735, at a maximum of 1.25% at 29 days.

However, in this field dissipation study metolachlor and its major degradates CGA-51202 and CGA-50720 were analyzed for as well as the degradates CGA-40172 and CGA-40919, which were only detectable but not quantifiable in the above referenced aerobic metabolism study. No explanation was provided by the registrant as to why the other major degradates listed above that were isolated in the aerobic metabolism study were not also included as standards to determine their environmental fate in the field dissipation study.

4. Field soil samples were stored frozen for up to 698 days prior to extraction; however, the stability of metolachlor and its degradates in the soil samples could not be confirmed because the available storage stability data were too variable. In a storage stability experiment conducted with soil taken from the control plot at the test site, soil samples (6-inch increments from depths of 6 inches to 60 inches) were fortified with metolachlor and the degradates CGA-51202, CGA-40172, and CGA-40919 at 1.0 and 5.0 ppm, then stored frozen for up to 901 days. After 70 days of storage, recoveries ranged from 60.4 to 170.6% of the applied; after 538/539 days, recoveries ranged from 30.8 to 166.4%; and after 901 days, recoveries ranged from 44.9 to 149.7%

(Table 8). The wide variability in the storage stability data may have been due to the inability of the method to accurately determine metolachlor residues.

In addition, the storage stability of the degradate CGA-50720 in soil samples fortified at 1.0 ppm was investigated at only one interval; after 127 days of storage, 60.8-80.4% of the applied was recovered. The 127-day sampling interval is inadequate since the analytical method was not modified to isolate CGA-50720 until the 317-day field soil samples were analyzed (see Comment 5).

5. Prior to analysis of the 317-day soil samples, the analytical method was modified to recover a fourth degradate (CGA-50720). The method description for the soil extraction was not included in the report proper, but was included in the Protocol (Appendix A). The method summarized in this report is the final modified method.

In addition, although not specifically stated, it appears from the extraction dates (Table 7) that all relevant soil samples were reanalyzed using the modified method.

6. Pretreatment samples from the crop and bareground plots contained up to 0.12-0.13 ppm metolachlor (Tables 11 and 12); soil from the control plot contained up to 0.11 ppm CGA-40172 and 0.21 ppm CGA-50720 (Table 10).
7. Air and soil temperature data were obtained from the NOAA weather station at Ames, Iowa; the distance from the weather station to the test site was not reported. Rainfall data were collected at the test site from June 4 to September 30, 1987, then for the remainder of the study, the data were obtained from the weather station. It is preferable that meteorological data be taken at the test site.
8. The depth to the water table was 5-8 feet, and the slope of the field was <1 degree. Field maintenance practices of the treated plot during the study were not reported. The plots had been planted to oats prior to the study; no pesticides or fertilizers had been applied for 3 years prior to the study.
9. Soil characteristics were provided for depths up to 5 feet (Table 3).
10. Two soil samples from each sampling depth were spiked in the field at 1.0 ppm; samples were sealed, frozen with dry ice, and stored frozen (temperature not specified) for 56 days before analysis. Recoveries ranged from 70.0 to 129.3% (Table 9).
11. The study site was the Staples Farm; however, the photographs of the test plots presented in this study (Figure 4) are identical to those presented for the Key farm (Studies 2 and 3, this report).

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